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MEMORANDUM

TO: Chris Lichens, Remedial Project Manager
Site Cleanup Section 4, SFD-7-4

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) RF
Quality Assurance (QA) Program, MTS-3

FROM: Doug Lindelof, Data Review Task Manager SL for DL.
Region 9 Environmental Services Assistance Team (ESAT)

ESAT Contract No.: EP-W-06-041
Technical Direction Form No.: 00105042 Amendment 4

DATE: August 10, 2007

SUBJECT: Review of Analytical Data, **Tier 2**

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Cenco Refinery Omega Chem
Site Account No.:	09 BC LA02
CERCLIS ID No.:	CAD042245001
Case No.:	Not Provided
SDG No.:	IPK0785 and IPK0962
Laboratory:	Test America Analytical Testing Corp.
Analysis:	1,2,3-Trichloropropane (1,2,3-TCP) and n-Nitrosodimethylamine (NDMA)
Samples:	5 Water Samples (see Case Summary)
Collection Date:	November 7 and 8, 2006
Reviewer:	Santiago Lee, ESAT/Laboratory Data Consultants (LDC)

This report has been reviewed by the EPA TOM for the ESAT contract, whose signature appears above.

If there are any questions, please contact Rose Fong (QA Program/EPA) at (415) 972-3812.

Attachment

SAMPLING ISSUES: [ ] Yes [X] No

Data Validation Report – **Tier 2**

Case No.: 335392.FI.01
SDG No.: IPK0785 and IPK0962
Site: Cenco Refinery Omega Chem
Laboratory: Test America Analytical Testing Corp.
Reviewer: Santiago Lee, ESAT/LDC
Date: August 10, 2007

I. CASE SUMMARY

Sample Information

Samples:	IPK0785 = OC2-MW606-W-0-264, OC2-MW605-W-0-265, and OC2-MW603-W-0-266; IPK0962 = OC2-MW105-W-2-267 and OC2-MW105-W-0-268
Concentration and Matrix:	Low Concentration Water
Analysis:	1,2,3-TCP (GC/MS) and NDMA (GC/MS/MS CI)
SOW:	EPA Methods 524.2 and 1625 Modified
Collection Date:	November 7 and 8, 2006
Sample Receipt Date:	November 7 and 8, 2006
Extraction Date:	November 12 and 16, 2006
Analysis Date:	November 14,, 15, and 17, 2006

Field QC

Field Blanks (FB):	OC2-MW105-W-2-267 (1,2,3-TCP only)
Trip Blanks (TB):	Not Provided
Equipment Blanks (EB):	Not Provided
Background Samples (BG):	Not Provided
Field Duplicates (D1):	Not Provided

Laboratory QC

Method Blanks & Associated Samples:

6K12038-BLK1:	(NDMA) All samples
C6K1602-BLK1:	(1,2,3-TCP) All samples

Tables

1B: Data Qualifier Definitions for Organic Data Review

Sampling Issues

None.

Additional Comments

As directed by the TOM, a Tier 2 validation (i.e., review all QC results and calibrations, minus calculation check) was performed.

For the NDMA analysis, decafluorotriphenylphosphine (DFTPP) was not analyzed. Since NDMA is analyzed by the chemical ionization (CI) technique, no adverse effect is expected.

For the 1,2,3-TCP analysis, 4-bromofluorobenzene (BFB) was not analyzed. Since 1,2,3-

TCP is analyzed by the selected ion monitoring (SIM) technique, no adverse effect is expected.

This report was prepared in accordance with the following documents:

- ESAT Region 9 Standard Operating Procedure 901, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Volatile and Semivolatile Data Packages*;
- EPA Method 524.2, *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*, Revision 4.1, 1995;
- EPA Method 1625C, *Semivolatile Organic Compounds by Isotope dilution GC/MS*, June 1989; and
- *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, October 1999.

II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

	Parameter	Acceptable	Comment
1.	Holding Time/Preservation	Yes	
2.	GC/MS and GC Performance	Yes	
3.	Initial Calibration	Yes	
4.	Continuing Calibration	Yes	
5.	Laboratory Blanks	No	A
6.	Field Blanks	N/A	
7.	Surrogate (Method 524.2)	No	D
8.	Labeled Compound (Method 1625)	No	E
9.	Matrix Spike/Matrix Spike Duplicates	No	C
10.	Laboratory Control Samples/Duplicates	Yes	
11.	Internal Standard	Yes	
12.	Compound Identification	Yes	
13.	Compound Quantitation	No	B
14.	System Performance	Yes	
15.	Field Duplicate Sample Analysis	N/A	

N/A = Not Applicable

III. VALIDITY AND COMMENTS

A. The following result is qualified as nondetected and estimated due to method blank contamination and is flagged "UJ" in the attached .xls file.

- NDMA in sample OC2-MW606-W-0-264

NDMA was found in method blank 6K12038-BLK1 at a concentration of 0.00053 ug/L. The NDMA result in sample OC2-MW606-W-0-264 is considered nondetected and estimated (UJ) and the reporting limit has been raised according to blank qualification rules presented below.

No positive results are reported unless the concentration of the compound in the sample exceeds 5 times the amount in any associated blank. If the sample result is greater than the reporting limit, the reporting limit is raised to the sample result and reported as nondetected. If the sample result is less than the reporting limit, the result is reported as nondetected at the reporting limit.

A laboratory method blank is laboratory reagent water or baked sand analyzed with all reagents, deuterated monitoring compounds, and internal standards and carried through the same sample preparation and analytical procedures as the field samples. The laboratory method blank is used to determine the level of contamination introduced by the laboratory during analysis.

B. The laboratory reported the NDMA sample reporting limit as 0.0020 ug/L. No NDMA was detected above this reporting limit. However, the area for the low standard of initial calibration is only 1059 (see attached quantitation report, p. 26 in data package). In the reviewer's professional judgment, the sample reporting limit should be raised to 0.01 ug/L; non-detected sample results are reported as 0.01U in the attached .xls file.

C. Matrix spike/matrix spike duplicate recoveries for 1,2,3-TCP in QC samples OC2-MW105-W-0-268-MS and OC2-MW105-W-0-268-MSD did not meet the laboratory QC limit, as shown below.

Analyte	C6K1602-MS1 % Recovery	C6K1602-MSD1 % Recovery	QC limit % Recovery
1,2,3-TCP	150	148	50-130

Results obtained may indicate poor laboratory technique or matrix effects which may interfere with analysis. Since 1,2,3-TCP is not detected in the samples, no adverse effect on data quality is expected.

D. For the 1,2,3-TCP analysis, the laboratory did not spike the samples, QC samples, and method blank with a surrogate (see Method 524.2 Sections 3.2, 7.5, 11.1.2, and 12.1.1 and Table 1). Consequently, the extraction efficiency (surrogate recovery) cannot be evaluated. The 1,2,3-trichloropropane-d5 spiked by the laboratory was used as an internal standard.

E. For the NDMA analysis, the laboratory did not spike the samples, QC samples, and method blank with a labeled compound (i.e., surrogate; see Method 1625C Sections 6.8, 10.2.1.3, and 10.2.3.2 and Figure 4). Consequently, the extraction efficiency (surrogate recovery) cannot be evaluated. The NDMA-d6 spiked by the laboratory was used as an internal standard.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR ORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999.

U The analyte was analyzed for but was not detected above the reported sample quantitation limit.

L Indicates results which fall below the Contract Required Quantitation Limit. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

FieldID	LabSampleID	Analyte	Result	Units	RL	Validity	Comment
OC2-MW606-W-0-264	IPK0785-01	N-Nitrosodimethylamine		UG/L	0.01	UJ	ABE
OC2-MW605-W-0-265	IPK0785-02	N-Nitrosodimethylamine		UG/L	0.01	U	BE
OC2-MW603-W-0-266	IPK0785-03	N-Nitrosodimethylamine		UG/L	0.01	U	BE
OC2-MW606-W-0-264	IPK0785-01	123-Trichloropropane (123-TCP)		NG/L	5	U	D
OC2-MW605-W-0-265	IPK0785-02	123-Trichloropropane (123-TCP)		NG/L	5	U	D
OC2-MW603-W-0-266	IPK0785-03	123-Trichloropropane (123-TCP)		NG/L	5	U	D
6K12038-BLK1	6K12038-BLK1	N-Nitrosodimethylamine	0.00053	UG/L	0.01		BE
6K12038-BS1	6K12038-BS1	N-Nitrosodimethylamine	0.00857	UG/L	0.01		BE
6K12038-BS2	6K12038-BS2	N-Nitrosodimethylamine	0.00207	UG/L	0.01		BE
6K12038-MS1	6K12038-MS1	N-Nitrosodimethylamine	0.00794	UG/L	0.01		BE
6K12038-MSD1	6K12038-MSD1	N-Nitrosodimethylamine	0.00792	UG/L	0.01		BE
C6K1602-BLK1	C6K1602-BLK1	123-Trichloropropane (123-TCP)		NG/L	5	U	D
C6K1602-BS1	C6K1602-BS1	123-Trichloropropane (123-TCP)	4.69	NG/L	5		D
C6K1602-MS1	C6K1602-MS1	123-Trichloropropane (123-TCP)	75	NG/L	5		D
C6K1602-MSD1	C6K1602-MSD1	123-Trichloropropane (123-TCP)	73.9	NG/L	5		D

FieldID	LabSampleID	Analyte	Result	Units	RL	Validity	Comment
OC2-MW105-W-0-268	IPK0962-02	N-Nitrosodimethylamine		UG/L	0.01	U	BE
OC2-MW105-W-2-267	IPK0962-01	123-Trichloropropane (123-TCP)		NG/L	5	U	D
OC2-MW105-W-0-268	IPK0962-02	123-Trichloropropane (123-TCP)		NG/L	5	U	D
6K12038-BLK1	6K12038-BLK1	N-Nitrosodimethylamine	0.00053	UG/L	0.01		BE
6K12038-BS1	6K12038-BS1	N-Nitrosodimethylamine	0.00857	UG/L	0.01		BE
6K12038-BS2	6K12038-BS2	N-Nitrosodimethylamine	0.00207	UG/L	0.01		BE
6K12038-MS1	6K12038-MS1	N-Nitrosodimethylamine	0.00794	UG/L	0.01		BE
6K12038-MSD1	6K12038-MSD1	N-Nitrosodimethylamine	0.00792	UG/L	0.01		BE
C6K1602-BLK1	C6K1602-BLK1	123-Trichloropropane (123-TCP)		NG/L	5	U	D
C6K1602-BS1	C6K1602-BS1	123-Trichloropropane (123-TCP)	4.69	NG/L	5		D
C6K1602-MS1	C6K1602-MS1	123-Trichloropropane (123-TCP)	75	NG/L	5		CD
C6K1602-MSD1	C6K1602-MSD1	123-Trichloropropane (123-TCP)	73.9	NG/L	5		CD

Quantitation Report (Not Reviewed)

Data File : C:\MSDCHEM\1\DATA\06NOV14\NNA001.D Vial: 2
Acq On : 14 Nov 2006 10:44 am Operator: DF/AI
Sample : 1 PPB Std.# 6060243 Inst : gcms37
Misc : n-Nitrosamines Water ICAL Multiplr: 1.00
MS Integration Params: RTEINT2.P
Quant Time: Nov 14 14:29:32 2006 Quant Results File: C6K14NWA.RES

Quant Method : C:\MSDCHEM\1\METHODS\C6K14NWA.M (RTE Integrator)
Title : Nitrosamine Water ICAL 11/14/06, Preextraction IS
Last Update : Tue Nov 14 14:29:13 2006
Response via : Initial Calibration
DataAcq Meth : C6J31NWA

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) NDMA-D6	10.77	81	2030	10.00	PPB	0.00
4) NDPA-D14	15.69	145	2859	10.00	PPB	0.00
Target Compounds						Qvalue
2) NDMA	10.70	92	1059	1.76	PPB	98
3) NDEA	13.21	120	967	2.26	PPB	97
5) NDPA	15.65	148	851	1.65	PPB	98
6) NPYR	17.06	118	934	2.28	PPB	94

(#) = qualifier out of range (m) = manual integration (+) = signals summed
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